ECBC-TR-###

Domestic Preparedness Program
Evaluation Of The Nextteq Civil Defense Kit^{tm}
Against Chemical Warfare Agents
Summary Report

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May 2004

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REPORT DOCUMENTATION PAGE			*Form Approved OMB No. 0704-0188*
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1. AGENCY USE ONLY *(Leave Blank)*	2. REPORT DATE May ***2004***	3. REPORT TYPE AND DATES COVERED ***Final; 03 Aug – 03 Oct***	
4. TITLE AND SUBTITLE DOMESTIC PREPAREDNESS PROGRAM EVALUATION OF THE NEXTTEQ CIVIL DEFENSE KIT™ AGAINST CHEMICAL WARFARE AGENTS, SUMMARY REPORT			5. FUNDING NUMBERS None
6. AUTHOR (S) Longworth, Terri L.; Matthews, Robin L.; Baranoski, John M			
7. PERFORMING ORGANIZATION NAME (S) AND ADDRESS (ES) TECH DIR, ECBC, APG, MD 21010-5424, ATTN: AMSRD-ECB-RT-AT.			8. PERFORMING ORGANIZATION REPORT NUMBER ECBC–TR-
9. SPONSORING/MONITORING AGENCY NAME (S) AND ADDRESS (ES) TECH DIR, ECBC, APG, MD 21010-5424, ATTN: AMSRD-ECB-ENH.			10. SPONSORING/MONITORING AGENCY REPORT NUMBER
11. SUPPLEMENTARY NOTES			
12a. DISTRIBUTION/AVAILABILITY STATEMENT Approved for public release; distribution is unlimited.			12b. DISTRIBUTION CODE
13. ABSTRACT (Maximum 200 words) This report characterizes the chemical warfare (CW) agent detection characteristics of the commercially available NEXTTEQ Civil Defense Kit™. The kit uses colorimetric detection tubes to detect the presence or absence of chemical warfare agents. The system was tested against HD, GB, and GA vapor under various conditions. This report provides the emergency responders concerned with CW agent detection and verification an overview of the capabilities of the Civil Defense Kit™.			
14. SUBJECT TERMS HD Chemical Warfare Agent (CWA) Detection Colorimetric Tubes GB Chemical Warfare (CW) agent vapor testing Detector Tubes GA Detector testing Interference testing			15. NUMBER OF PAGES 29 16. PRICE CODE
17. SECURITY CLASSIFICATION OF REPORT UNCLASSIFIED	18. SECURITY CLASSIFICATION OF THIS PAGE UNCLASSIFIED	19. SECURITY CLASSIFICATION OF ABSTRACT UNCLASSIFIED	20. LIMITATION OF ABSTRACT UL

NSN 7540-01-280-5500

Standard Form 298 (Rev. 2-89)
Prescribed by ANSI Std. Z39-18
298-102

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PREFACE

The work described herein was authorized under the Expert Assistance (Equipment Test) Program for the U.S. Army Edgewood Chemical Biological Center (ECBC) Homeland Defense Business Unit. This work was started in August 2003 and was completed in January 2004.

The use of either trade or manufacturers' names in this report does not constitute an official endorsement of any commercial products. This report may not be cited for purposes of advertisement.

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CONTENTS

FIGURES

TABLES

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DOMESTIC PREPAREDNESS PROGRAM
EVALUATION OF THE NEXTTEQ CIVIL DEFENSE KIT™
AGAINST CHEMICAL WARFARE AGENTS
SUMMARY REPORT

1. INTRODUCTION

The Department of Defense (DOD) formed the Domestic Preparedness (DP) Program in 1996 in response to Public Law 104-201. One of the objectives is to enhance federal, state, and local capabilities to respond to Nuclear, Biological and Chemical (NBC) terrorism incidents. Emergency responders who encounter either a contaminated or a potentially contaminated area must survey the area for the presence of either toxic or explosive vapors. Presently, the vapor detectors commonly used are not designed to detect and identify chemical warfare (CW) agents. Little data are available concerning the ability of these commonly used, commercially available detection devices to detect CW agents. Under the DP Expert Assistance (Test Equipment) Program, the U.S. Army Edgewood Chemical Biological Center (ECBC) Homeland Defense Business Unit, formerly known as the U.S. Army Soldier and Biological Chemical Command (SBCCOM) continues to address this need. The Applied Test Team (ATT) at ECBC, Aberdeen Proving Ground, Maryland, performed the testing. ATT is tasked with providing the necessary information to aid authorities in the selection of detection equipment applicable to their needs.

Reports of the instrument evaluations are posted in the Homeland Defense website (http://www.ecbc.army.mil/hld) for public access. Instruments evaluated and reported since 1998 include:

- MiniRAE plus from RAE Systems, Incorporated (Sunnyvale, CA)
- Passport II Organic Vapor Monitor from Mine Safety Appliance Company (Pittsburgh, PA)
- PI-101 Trace Gas Analyzer from HNU Systems, Incorporated (Newton, MA)
- TVA 1000B Toxic Vapor Analyzer (PID and FID) from Foxboro Company (Foxboro, MA).
- Draeger Colorimetric Tubes (Thioether and Phosphoric Acid Ester) from Draeger Safety, Incorporated (Pittsburgh, PA)
- Photovac MicroFID detector from Perkin-Elmer Corporation (Wellesley, MA)
- MIRAN SapphIRe Air Analyzer from Foxboro Company (Foxboro, MA)
- MSA Colorimetric Tubes (HD and Phosphoric Acid Ester) from Mine Safety Appliances Company (Pittsburgh, PA)
- M90-D1-C Chemical Warfare Detector from Environics OY, Finland
- APD2000 Detectors from Environmental Technologies Group, Incorporated (Baltimore, MD)
- SAW MiniCAD mkII from Microsensor Systems, Incorporated (Apopka, FL)
- UC AP2C Monitor from Proengin Incorporated, France
- ppbRAE Photo-Ionization Detector from RAE Systems, Incorporated (Sunnyvale, CA)

- SABRE2000 detector from Barringer Technologies, Incorporated (Warren, NJ)
- CAM (Type L) from Graseby Dynamics Ltd., United Kingdom
- VaporTracer System from Ion Track Instruments, Incorporated (Wilmington, MA)
- HAZMATCAD from Microsensor Systems, a Sawtek Company (Apopka, FL)
- GC-MS/FPD with Dynatherm System from Agilent (Columbia, MD)
- Scentoscreen GC from Sentex Systems, Incorporated (Fairfield, NJ)
- RAID-M from Bruker Saxonia Analytik GmbH (Leipzig, Germany)
- IMS2000 from Bruker Daltonics GmbH (Switzerland)
- Travel*IR* from SensIR Technologies (Danbury, CT)

In 2003, the evaluations continued using the NEXTTEQ Civil Defense Kit™ that was loaned to the DP program by the manufacturer. In exchange, the detection kit was evaluated under the DP protocol and the manufacturer was permitted to take data during the evaluations. This report summarizes the evaluation of the NEXTTEQ Civil Defense Kit™.

2. OBJECTIVE

The objective of this report is to assess the capability and general characteristics of the NEXTTEQ Civil Defense Kit™ to detect CW agent vapors. The intent is to provide the emergency responders concerned with CW agent detection an overview of the detection capabilities of this detection kit.

3. SCOPE

The scope of this DP evaluation is to characterize the CW agent vapor detection capability of the NEXTTEQ Civil Defense Kit™. The agents used included tabun (GA), sarin (GB), and mustard (HD). These were chosen as representative threat CW agents. Test procedures follow the established DP Detector Test and Evaluation Protocol described in the Phase 1 Test Report[1]. The test concept was as follows:

a. Determine the Minimum Detectable Level (MDL) where repeatable detection readings are achieved for each selected CW agent. The current military Joint Services Operational Requirements (JSOR)[2] served as a guide for detection sensitivity objectives.

b. Investigate the effects of humidity and temperature on system performance.

c. Observe the effects of potential interfering substances upon system performance both in the laboratory and in the field.

4. EQUIPMENT AND TEST PROCEDURES

4.1 System Description

The Civil Defense Kit™ is manufactured by NEXTTEQ, LLC (Tampa, FL). Their website is located at www.NEXTTEQ.com. The Civil Defense Kit was loaned to the DP Program for inclusion in the 2003 detector evaluations. According to the manufacturer's operating manual[3], the Civil Defense Kit™ is a hand held detection system capable of detecting the presence or absence of a wide range of chemical warfare agents (CWA). The manufacturer states that the three main components of the kit are the Chemical Agents Detector (CAD) Tubes Set, the Civil Defense Manifold, and a pumping system. The kit also provides accessories for remote sampling and replacement parts for expendables.

The CAD Tubes Set is a disposable single use packaged set of glass detector tubes that uses unique chemical and biochemical colorimetric reagents to detect and identify CW agents. The CAD Tubes Set weighs 0.25 oz and contains five colorimetric detection tubes. The set of tubes is 4" by 4 7/8" by ¼" in dimension. Three out of the five types of tubes in the set were evaluated for their ability to detect the CW agents GA, GB and HD. The tubes evaluated were the Phosphoric Acid Esters (PAE) tube for nerve agent detection, and both the HD, HN tube and the HD only tube for blister agent detection. The manufacturer states that the PAE tube identifies the nerve agents GA, GB, GD, VX, GP, and GF while the blister agent tubes identify H, HD, and HN. The other two tubes in the tubes set are the L tube for detecting lewisite and the CK, AC, DP, CG tube for detecting cyanogen chloride, hydrogen cyanide, diphosgene, and phosgene, respectively. The operational temperature range for the detector tubes is given as 0°C to 40°C (32°F to 104°F) with relative humidity conditions between 10% and 80%. The specification for storage and transport temperatures is up to 25°C (77°F). However, tubes were transported and stored at ambient temperatures during the evaluation. The manufacturer of the individual tubes is Mine Safety Appliances Company and the part numbers of the tubes tested in this report are as follows: PAE tube part number 10007654-msa, HD tube part number 10007653-msa, and HD, HN tube part number 10007652-msa.

The second component of the kit is the Civil Defense Manifold that holds the CAD Tubes Set. The manifold consists of a series of preset limited orifices to deliver a specific volume of airflow through each tube during vapor sampling. The manifold has a pistol handle grip, weighs 7 oz, and has dimensions of 5¼" by 1 3/8" by 1". Shown on the manifold is the chemical reaction color change comparison chart needed to check for the respective positive response of each tube.

The third component of the Civil Defense Kit™ is the pumping system that attaches to the manifold to draw a preset volume of air through the manifold for tube sampling. The kit includes the following three pump options for the user: the NX2000 electric pump, the NXP500 hand piston pump, and the NXV2000 venturi pump.

With these pump options, the power requirement, size, and weight of the Civil Defense Kit™ system will vary depending on the pumping system used. The hand piston pump is manually operated and requires no power supply. The electric pump uses a standard 120V AC outlet and weighs approximately 2 lbs. The venturi pump attachments require a gas source such as a firefighter's breathing tank. In all cases the kit is an easily manageable hand held detection system. Figure 1 is a picture from the manufacturer of the Civil Defense Kit™ showing the manifold attached to the three different pumping system options. Due to time constraints and for test consistency, only the electric pump was used during the DP evaluations of the system.

Figure 1. NEXTTEQ Civil Defense Kit™

4.2 Tube Sampling Procedures

Operating procedures were followed according to the operating instruction manual. In addition to the manual, the Civil Defense Kit™ includes for ease of use a one page laminated specifications and operating instruction sheet that also describes the chemical reaction color change for each tube and includes cross interferences guidance. The respective step-by-step sampling procedure for each tube type with diagrams is presented on this page. No daily instrument calibration is required by the manufacturer to place the kit into operation. The electric pump included in the kit is pre-calibrated at the factory to pull 5 liters per minute through the manifold. The manifold divides the flow pulled through each tube by using the manufacturer's preset orifices to deliver the specific volume needed for each tube. The flows of the pump and each of the five tube inlets of the manifold were confirmed prior to testing and rechecked periodically throughout testing. If the flows dropped below 30% of the starting values, the pump was exchanged to correct the flows.

Figure 2 shows the configuration of the Civil Defense Kit™ system as tested with the main components labeled. The CAD Tubes Set (1) requires snapping off the glass tips at each end of the tubes before attaching to the Civil Defense Manifold (2). This is accomplished by holding the pistol handle (2a) of the manifold and inserting the tubes set into the small holes of the tube opener on the manifold (2b) and breaking, then turning the set upside down to break the opposite side. Prior to vapor sampling, the PAE tube requires the ampoule on the white layer side to be crushed with the ampoule crushing pin (2c) that is stored on the manifold. After crushing the ampoule, the solution from the ampoule must be manually shaken down to impregnate the white layer of the PAE tube. The other tubes in the tubes set do not require activation prior to sampling. The tubes set is then inserted into the tube nipples of the manifold (2d) with the arrows on the tubes set pointing down into the manifold.

The manifold was attached to the NX2000 pump (3) with the 1 ft hose assembly (4) and the quick-attach decontamination filter (5) during the DP tests. The system begins vapor sampling after the run button is pressed on the pump. The pump is preset by the manufacturer to run for 3.5 minutes while pulling 5 liters per minute for sampling. The directions require a 2 minute wait after sampling before continuing with the respective tube activations.

Figure 2. NEXTTEQ Civil Defense Kit™ configuration

After sampling and waiting 2 minutes, the HD only tube requires no activation. A color change of the substrate layer from yellow to orange will occur within the given timeframe to indicate a positive response to mustard. The HD, HN tube requires activation by crushing its ampoule and shaking the liquid into the substrate layer. The light yellow HD, HN tube will change to show a blue ring to indicate a positive response. The activation of the PAE tube requires the second ampoule at the yellow layer end of the tube to be crushed. The liquid from the ampoule must be shaken down through the yellow layer into the white layer for tube activation. The manufacturer recommends, if necessary, to reinsert the tubes set from the side of the crushed ampoules back into the manifold and run the pump for 10 seconds to ensure the solutions from the ampoules impregnate the layers. The white layer of the PAE tube should remain white to indicate presence of a nerve agent. Otherwise the white layer of the PAE tube will turn yellow indicating no nerve agent is present.

According to the manufacturer's instructions, 2 minutes after tube activations the colors of the tube layers should be compared with the chemical reaction color chart shown on the instruction sheet or on the front of the manifold. During the DP evaluations the tubes were activated as directed and the detection reaction process yielding color development was observed. A positive response indicating agent detection required the presence of the appropriate color development in the respective tube within 2 minutes after tube activations.

4.3 Agent Vapor Challenge

The agent challenges were conducted using the Multi-Purpose Chemical Agent Vapor Generation System[4] using Chemical Agent Standard Analytical Reference Material (CASARM) grade or the highest purity CW agents available. The vapor generator system permits testing of an instrument with humidity and temperature-conditioned air without agent vapor before challenging it with similarly conditioned air containing the CW agent vapor. Blank tests were run on the tubes, as references, exactly as the agent tests by sampling the vapor generator's conditioned air without agent at the respective temperature and humidity conditions.

Agent challenge begins when the solenoids of the vapor generation system are energized to switch the air streams from conditioned air only to similarly conditioned air containing the agent. The agent vapor was drawn directly from the vapor generator into the prepared tubes by the kit's pump. Due to the nature of the vapor generator to provide only one agent vapor at a time, the tubes were tested individually and not as a tube set. For the DP evaluations, the manufacturer provided a restrictor that was designed to simulate each different tube of the tube set. These restrictors were placed on the respective ports of the manifold for the tubes not being tested at the time. This ensured that the sampling flows through each of the manifold ports equaled the sampling flows seen when using the tube set in its entirety. The air flows of the tubes in the tubes set and the tubes with the restrictors in the empty ports were demonstrated to be equal.

The tubes were tested with the CW agents GA, GB, and HD at several concentration levels at ambient temperature and relative humidity to determine the minimum detectable level (MDL) of the respective tube for each agent. Three tubes with positive color responses were required to determine the MDL for each tube type.

The tubes were then tested at the determined MDL concentrations under humidity and temperature extremes. The agent concentrations were raised or lowered to obtain, if possible, three tubes with positive color responses under each condition tested. The tubes were tested at both <5% RH and >80% RH in ambient temperatures to observe potential humidity effects. Testing at 5°C, the temperature recommended by the manufacturer, assessed the effects of low temperature. Although HD freezes at approximately +15°C, the calculated HD volatility of 92 mg/m^3 at 0°C easily produces a vapor concentration higher than the 2 mg/m^3 Joint Service Operational Requirement (JSOR) detection criteria allowing the instrument to be evaluated against HD down to 5°C. The effects of high temperatures were assessed by testing at +40°C per the manufacturer's specifications.

In order to investigate the effects of temperature on the performance of the Civil Defense Kit™, the system along with the tubes were placed in the environmental temperature chamber for temperature conditioning for a minimum of 2 hours prior to testing. Blanks, agent challenges, and color development were performed in the temperature chamber to assure a consistent temperature environment during the testing process.

4.4 Agent Vapor Quantification

The generated agent vapor concentrations were analyzed independently and are reported in the data tables. The vapor concentration was quantified by utilizing the manual sample collection methodology[5] using the Miniature Continuous Air Monitoring System (MINICAMS®) manufactured by O. I. Analytical, Inc. (Birmingham, AL). The MINICAMS® is equipped with a flame photometric detector (FPD), and was operated in either phosphorus mode for the GA and GB agents or sulfur mode for HD.

The MINICAMS® normally monitors air by collection through sample lines then adsorbing the CW agent onto the solid sorbent contained in a glass tube referred to as the pre-concentrator tube (PCT). The PCT is located after the MINICAMS® inlet. The concentrated sample is periodically heat desorbed into a gas chromatographic capillary column for subsequent separation, identification, and quantification. For manual sample collection, the PCT was removed from the MINICAMS® during the sampling cycle and connected to a measured suction source to draw the vapor sample from the agent generator. The PCT was then re-inserted into the MINICAMS® for analysis. This "manual sample collection" methodology eliminated potential loss of sample along the sampling lines and the inlet assembly when the MINICAMS® was used as an analytical instrument. The calibration of the MINICAMS® was performed daily using the appropriate standards for the agent of interest. The measured mass equivalent (derived from the MINICAMS chromatogram) divided by the total volume (flow rate multiplied by time) of the vapor sample drawn through the PCT produced the sample concentration that converts into milligrams/cubic meter.

4.5 Laboratory Interference Tests

The laboratory interference tests were designed to assess the effect on the detection reaction process of the tubes from potential interfering substances. The substances were chosen based on the likelihood of their presence during an emergency response by first responders. The tubes were tested against 1% of the headspace concentrations of gasoline, jet propulsion fuel (JP8), diesel fuel, household chlorine bleach, floor wax, Aqueous Film Forming Foam (AFFF, used for fire fighting), Spray 9™ household cleaner, vinegar, Windex™ window cleaner, toluene and antifreeze vapors. They were also tested against 25 ppm NH_3 (ammonia).

If the tubes showed no response after sampling the vapor of the potential interfering substance, requiring that the tube respond as a blank, testing was performed using CW agent plus the substance to assess the CW agent detection capability in the presence of the potential interfering substance.

To generate the vapor concentration of the potential interfering substances, a dry air stream carried the headspace vapor of the substance by sweeping either over the liquid in a tube or through the liquid in a bubbler. In order to produce the 1% concentration of interferent test mixture, 30 milliliters/minute (mL/min) of the vapor saturated air was diluted to three liters/minute (L/min) with the conditioned air at ambient temperature and humidity. The 25 parts per million (ppm) ammonia was derived by proper dilution of a stream from an analyzed 1% NH_3 vapor (10,000 ppm) compressed gas cylinder diluted with the appropriate amount of the conditioned air. Two tubes were exposed to the potential interfering substance vapors and, if they exhibited no interference, a third tube was tested against the CW agent plus the interferent.

Due to time constraints, only HD and one nerve agent, GB, were used during the laboratory interference tests. For the tests that included CW agent, the interferent test gas mixture was prepared similarly. The resultant stream of three L/min of CW agent vapor was used as a dilution stream to blend in with the 30 mL/min of the substance vapor flow to obtain the desired 1% mixture of the substance vapor in the presence of CW agent concentration.

4.6 Field Interference Tests

The Civil Defense Kit™ was tested outdoors in the presence of common potential interferents such as the vapors from gasoline, diesel fuel, jet propulsion fuel (JP8), kerosene, Aqueous Film Forming Foam (AFFF, used for fire fighting), household chlorine bleach, and insect repellent. Vapor from a 10% calcium hypochlorite solution (HTH slurry, a chlorinating decontaminant for CW agents), engine exhausts, burning fuels, and other burning materials were also tested. The objective was to assess the ability of the system to withstand outdoor environments and to resist false positive responses when exposed to the selected substances. In these tests, no CW agent was present.

The field tests were conducted outdoors at M-Field of the Edgewood Area, Aberdeen Proving Ground, for three days during September through October 2003. These experiments involved open containers, truck engines, and fires producing smoke plumes, which were sampled by the detectors at various distances downwind. The tubes were exposed to either the smoke or fume test plumes to achieve moderate concentrations (e.g. 1-5 ft for vapor fumes and 4-15 ft for smokes).

The flow of the pump and manifold ports were checked prior to beginning the field tests and periodically throughout each test day. Blank tests of the three different detector tubes were performed in the 'clean' field environment to assure that the tubes did not yield false positives prior to exposures to the potential field interfering substances.

Two of each type of tubes evaluated (PAE, HD only, and HD, HN) were exposed against each potential field interferent for the 3.5 minutes sampling time of the kit. The only exception was for the doused wood fire smoke where the fire could only be doused once; consequently only one of each tube type was exposed. During the field tests, the manifold held the three tube types and restrictors were placed on the other two ports so the three tubes were exposed and sampled simultaneously.

5. RESULTS AND DISCUSSION

5.1 Minimum Detectable Levels (MDL)

The MDL values for the Civil Defense Kit™ tube types tested, HD only, HD, HN, and Phosphoric Acid Esters (PAE), are shown in Table 1. The MDL values represent the lowest CW agent concentration exposure to produce positive color development consistently for three trials. Concentration units are shown in both milligrams per cubic meter (mg/m^3) and ppm. The MDL was established at ambient temperatures (20-25^{o}C) and mid-level relative humidity (48-55% RH). For comparison, the current military JSOR requirements for CW agent sensitivity for point detection alarms, the U.S. Army's established values for Immediate Danger to Life or Health (IDLH), and the Airborne Exposure Limit (AEL), are also listed in Table 1. Army Regulation (AR) 385-61 is the source for the IDLH and AEL values for GA and GB, and the AEL value for HD. The AR 385-61 does not establish an IDLH for HD due to concerns over carcinogenicity.

The blister agent tubes (HD only and HD, HN) were able to detect HD concentrations close to the JSOR level. The HD, HN tube showed a blue ring for positive detection at 2.2 mg/m^3 and the HD only tube showed an orange ring for positive detection at 3.0 mg/m^3. The Phosphoric Acid Esters (PAE) tubes were able to detect GA and GB at approximately the JSOR and IDLH values. The PAE tube remained white after activation to show positive detection for GA at 0.2 mg/m^3 and positive detection for GB at 0.1 mg/m^3.

The tubes were unable to detect GA, GB, or HD at the AEL levels. At values below the given MDL for the HD only and HD, HN tubes, the tube layer remained yellow or did not consistently turn the appropriate color. At values below the given MDL for the PAE tubes, the tube layer turned yellow or partially yellow indicating no agent present. It should be noted that there was difficulty in color development determination and conflicting opinions occurred among several observers regarding whether or not the results were positive on exposures at the threshold detection concentration levels.

The complete cycle time for the Civil Defense Kit™ includes the 3.5 minutes sampling time plus the required 2 minutes wait after vapor sampling before tube activations plus the actual time required by the operator to break the ampoules of each tube for activation plus an additional 2 minutes wait for color development. The time required to break the ampoules depended on the operator. Also it appeared that some of the tubes were harder to break than others. On average it required 5-60 seconds to break the ampoules and shake the liquid onto the tube layer for tube activation.

Table 1. Minimum Detectable Level (MDL) at Ambient Temperatures and Mid-Level Relative Humidity for Civil Defense Kit™ Tubes

Agent and Tube	Concentration in milligrams per cubic meter, mg/m^3, with parts per million values in parentheses (ppm)			
	Civil Defense Kit™ Tube MDL	JSOR*	IDLH**	AEL***
HD with HD tubes	3.0 (0.45)	2.0 (0.30) in 120 sec	N/A	0.003 (0.0005) up to 8 hr
HD with HD, HN tubes	2.2 (0.33)	2.0 (0.30) in 120 sec	N/A	0.003 (0.0005) up to 8 hr
GA with PAE tubes	0.2 (0.03)	0.1 (0.02) in 30 sec	0.2 (0.03) up to 30 min	0.0001 (0.00002) up to 8 hr
GB with PAE tubes	0.1 (0.02)	0.1 (0.02) in 30 sec	0.2 (0.03) up to 30 min	0.0001 (0.00002) up to 8 hr

* Joint Service Operational Requirements for detectors.
** Immediate Danger to Life or Health values from the unclassified Army Regulation (AR) 385-61, Feb 1997, to determine level of CW protection. Personnel must wear either the full ensemble with SCBA for operations or full-face respirator for escape.
***Airborne Exposure Limit values from AR 385-61 to determine masking requirements. Personnel can operate for up to 8 hr unmasked.

5.2 Temperature and Humidity Effects

Tables 2 and 3 show the results of the Civil Defense Kit™ evaluations using the HD only and HD, HN tubes under various test conditions against HD. Tables 4 and 5 show the results of the Phosphoric Acid Esters (PAE) tubes under various test conditions against GA and GB, respectively. Humidity effects tests were conducted at ambient temperatures and approximately 5%, 50%, and >80% RH. An attempt was made to test the tubes within the given operational range at temperature extremes of 5°C and 40°C.

The concentrations used to determine the temperature and humidity effects were based on the previously determined MDLs. Positive detection response is defined as three consistent responses in three independent trials for the agent at the temperature and RH specified. The Tables present a column for the tube color development response and indicates either a positive or negative agent detection. The intensity of color change and broadness of the color band of the respective tubes were demonstrated to increase as the agent concentration increased.

Table 2. Civil Defense Kit™ HD Tube Responses to HD Vapor Concentrations at Various Conditions

Average Conditions		HD Challenge Concentration		HD Only Tube Response
Temperature, °C	RH, %	mg/m³	ppm	
20-21	5	3.1	0.47	<1 mm orange ring, positive
		4.4	0.67	2 mm orange ring, positive
21-25	50-51	2.3	0.35	Slight discoloration, negative
		2.9	0.44	Discoloration, negative
		3.0	0.45	2 mm light orange ring
		4.1	0.62	2 mm orange ring
21-22	82-83	3.95	0.60	Yellow, negative
		7.76	1.18	Yellow, negative
5	50-52	3.97	0.57	Yellow, negative
		5.38	0.77	Yellow, negative
		6.21	0.89	Yellow, negative
40	46	1.60	0.26	6 mm orange ring, positive
		2.9	0.47	Entire tube dark orange, positive

Table 2 shows that the HD only tube demonstrated HD detection at the previously determined MDL at ambient temperature and low RH (5%) but showed no HD response in high humidity (82-83% RH) tests even at double the MDL concentration level. Cold temperature tests at 5°C yielded no HD response even at twice the MDL. In addition to testing the HD tubes conditioned in the cold temperature, HD tubes were entered into the 5°C chamber from room temperature and immediately tested. These tests also resulted in no HD detection at 5°C. High temperature tests at 40°C produced a beneficial effect on tube response. An immediate color change occurred at the MDL during agent exposure leading to orange color throughout the whole tube. At approximately half the MDL concentration, the positive orange color was still observed over 50 percent of the tube in the high temperature.

Table 3. Civil Defense Kit™ HD, HN Tube Responses to HD Vapor Concentrations at Various Conditions

Average Conditions		HD Challenge Concentration		HD, HN Tube Response
Temperature, °C	RH, %	mg/m³	ppm	
20-22	3-5	2.3	0.34	Slight discoloration*, negative
		3.2	0.49	1 mm blue ring, positive
20-21	50-51	1.0	0.15	Slight discoloration*, negative
		1.5	0.22	Discoloration, negative
		1.8	0.27	Discoloration**, negative
		2.0	0.30	1 mm blue ring***, yellow, negative
		2.2	0.33	1 mm blue ring
		4.1	0.62	1 mm blue ring
20-22	82-87	2.8	0.42	Slight discoloration**, negative
		3.95	0.60	<1 mm blue ring, positive
		7.76	1.18	2 mm blue ring, positive
5	50-52	3.97	0.57	Yellow, negative
		5.38	0.77	Yellow, negative
		6.21	0.89	Yellow, negative
40	46	1.6	0.26	Yellow****, negative
		2.6	0.42	<1 mm blue/gray ring**, positive

*Two out of three trials showed discoloration, the third trial was yellow, negative
**One of the trials showed a 1 mm blue ring, positive
***Two out of three trials showed 1 mm blue ring, the third trial was yellow, negative
****One of the trials showed a 1 mm gray ring

Table 3 shows that humidity and temperature affected the ability of the HD, HN tubes to detect HD consistently at the previously determined MDL. However,the tubes were able to produce a positve 1 mm blue ring for three trials at HD concentrations slightly above the MDL values during high and low RH, and high temperature tests. The tubes were unable to detect HD at 5°C. The liquid in the ampoules appeared to become slushy in the cold temperature. Therefore, tubes were placed on the pump to pull the liquid down to ensure that the tube layer was wetted. Tubes were also brought into the cold chamber from room temperature and immediately tested in the cold but no HD response was observed.

Table 4. Civil Defense Kit™ Phosphoric Acid Esters (PAE) Tube Responses to GA Vapor Concentrations at Various Conditions

Average Conditions		GA Challenge Concentration		Phosphoric Acid Esters (PAE) Tube Response
Temperature, °C	RH, %	mg/m³	ppm	
20	3	0.19	0.03	Very light yellow, negative
		0.29	0.04	2 white, 1 yellow, negative
		0.33	0.05	White, positive
20-21	48-51	0.03	0.004	Light yellow, negative
		0.10	0.015	1 white, 2 Yellow, negative
		0.15	0.022	1 white, 2 Yellow, negative
		0.20	0.030	White, positive
20-21	86-92	0.17	0.03	Yellow, negative
		0.28	0.04	2 half yellow, I yellow, negative
		0.44	0.07	1 white, 2 half yellow, negative
		0.63	0.09	White, positive
5	50	Blanks showed false positives, agent testing not completed		
40	38	Blanks showed false positives, agent testing not completed		

Table 4 shows that temperature and humidity had adverse effects on GA detection by the PAE tubes. The tubes required a concentration slightly higher than the MDL at low humidity (3% RH) for GA detection. At high humidity (86-92% RH) the GA concentration had to be increased from the MDL, 0.2 mg/m³, to 0.63 mg/m³ for positive results.

Testing at low temperature (5°C) resulted in the liquid of the tubes becoming sluggish. Blank tests were attempted before introducing agent and the blanks showed false positives. A false positive response indicates that the tube showed an agent detection response in the absence of CW agent. Per manufacturer's instructions, the pump was used to pull the liquid down to the layer to help with color development. Also, additional tubes were entered into the cold chamber from room temperature and immediately tested. Five blank PAE tubes were tested and failed at 5°C and 50% RH. The tubes remained white indicating agent detection instead of turning yellow indicating no agent during the testing of the blanks. Since false positives occurred, the tubes were not tested against GA at 5°C.

Similarly, agent testing of the PAE tubes with GA at 40°C was not completed when the PAE blanks demonstrated false positives in the temperature chamber. Additional tubes were entered into the hot temperature chamber from room temperature and immediately tested and still showed false positive responses. Testing of PAE blanks at 40°C yielded 6 white, false positives and one tube showing 50% white and one tube showing 25% white. The chamber temperature was lowered to 30°C and three out of five blanks tested showed false positives. However, two blanks tested at 37°C in a second temperature chamber worked properly. Investigations into the PAE blank failures indicated the possibility of some tube interference in the temperature chamber. However, the chamber in which these tests were conducted did not contain any chemical agents as proven by results of long sample times with negative detection of any GC peaks using the MINICAMS®. Additional temperature testing for the PAE tubes is required but was not possible due to time constraints.

Table 5. Civil Defense Kit™ Phosphoric Acid Esters (PAE) Tube Responses to GB Vapor Concentrations at Various Conditions

Average Conditions		GB Challenge Concentration		Phosphoric Acid Esters (PAE) Tube Response
Temperature, °C	RH, %	mg/m^3	ppm	
20	3	0.12	0.021	White, positive
20-21	48-55	0.012	0.002	1 white, 2 yellow, negative
		0.03	0.005	1 white, 2 yellow, negative
		0.08	0.013	1 white, 2 yellow, negative
		0.10	0.017	White
		0.16	0.027	White
		0.21	0.036	White
20	93	0.13	0.022	White, positive
5	50	Blanks showed false positives, agent testing not completed		
	0	Blanks were very light yellow/cream, negative		
		0.10	0.017	2 white, 1 yellow, negative
40	13	0.35	0.064	2 white, positive
40	26	0.56	0.103	1 white, 2 yellow, negative
40	44	0.98	0.180	2 yellow, negative

Table 5 shows that the PAE tubes showed no adverse effects at high or low humidity for GB detection. The PAE tubes were able to detect GB at the determined MDL at 3% RH and at 93% RH.

As previously discussed, the PAE tubes at 5°C and 50% RH gave white, false positive results. Further testing at 5°C and 0% RH yielded PAE blanks that were described as a creamy, very light yellow. Therefore, tubes were tested against GB at 5°C and 0% RH at 0.1 mg/m^3. The results were inconsistent and GB was only detected 2 out of 3 times. Distinguishing between white, cream and very light yellow was difficult for the operators in most tests at 5°C.

As previously discussed, the PAE tubes showed false positive responses at 40°C. However, further testing of blank tubes over several days correctly yielded 12 negative yellow responses. Therefore, the PAE tubes were tested against GB at 40°C. However, the results were inconsistent showing positive detection at 0.35 mg/m^3 while showing negative results at higher GB concentrations of 0.56 and 0.98 mg/m^3.

Due to time constraints, further investigation into the inconsistencies in PAE tubes could not be pursued. At this point, further temperature testing is required to make conclusive temperature evaluations for the PAE tubes.

5.3 Laboratory Interference Tests

The laboratory interference tests were conducted at ambient temperatures (20-29°C) and RH (43-53%). The three tube types (HD only, HD, HN, and PAE) were exposed to each potential interferent at 1% of saturation. If the tubes showed no response to the interferent after two trials, a third tube was exposed to the respective CW agent in the presence of the interferent. The Civil Defense Kit™ tubes were tested against both HD and GB using concentrations above the previously determined MDL.

Table 6 presents the results of exposing the tubes to several potential interferents without agent and in the presence of agent. None of the interferences tested caused any problems with any of the tubes. All of the tubes reacted as a blank, negative, when exposed to the potential interferent indicating no agent present.

Also, the tubes were able to detect the respective agent in the presence of the interferent. The HD only tubes and the HD, HN tubes were able to give positive HD response at HD concentrations between 3 and 6 mg/m^3 in the presence of the interferent. The PAE tubes demonstrated positive GB detection at 0.12-0.13 mg/m^3 GB concentrations in the presence of the interferents tested.

Table 6. Results of Laboratory Interference Tests of Civil Defense Kit™ HD only, HD, HN, and Phosphoric Acid Esters (PAE) Tubes at Ambient Conditions

Interferent	HD Only Tubes		HD, HN Tubes		Phosphoric Acid Esters Tubes	
	Interferent Only	Interferent plus HD at 3-6 mg/m^3	Interferent Only	Interferent plus HD at 3-7 mg/m^3	Interferent Only	Interferent plus GB at 0.12-0.13 mg/m^3
1% AFFF	Negative	Positive	Negative	Positive	Negative	Positive
1% Bleach	Negative	Positive	Negative	Positive	Negative	Positive
1% Diesel	Negative	Positive	Negative	Positive	Negative	Positive
1% Floor Wax	Negative	Positive	Negative	Positive	Negative	Positive
1% Gasoline	Negative	Positive	Negative	Positive	Negative	Positive
1% JP8	Negative	Positive	Negative	Positive	Negative	Positive
1% Spray 9™	Negative	Positive	Negative	Positive	Negative	Positive
1% Vinegar	Negative	Positive	Negative	Positive	Negative	Positive
1% Windex™	Negative	Positive	Negative	Positive	Negative	Positive
1% Antifreeze	Negative	Positive	Negative	Positive	Negative	Positive
1% Toluene	Negative	Positive	Negative	Positive	Negative	Positive
25 ppm Ammonia	Negative	Positive	Negative	Positive	Negative	Positive

5.4 Field Interference Tests

The results of the tube evaluations during the field test interferent exposures are presented in Table 7. The ambient temperature and relative humidity levels during these tests were in the range of 12-23°C (53-71°F) and 34-75% RH, with gentle to moderate winds.

Two tubes of each type (HD only, HD, HN, and PAE) were tested against the listed interferents except for the doused wood fire that was only doused and tested once. Also a third PAE tube was exposed to bleach vapor when one of the two tubes tested showed a false positive. The third tube showed a negative response to the bleach vapor. Blank tubes were successfully tested throughout the field test evaluations. The tubes were exposed to the field interferent for the preset pump pull of 3.5 minutes.

Pump and manifold flows were taken at the beginning and throughout the field test evaluations. The flows dropped slightly from continuous use of the pump and manifold during the two months of evaluations prior to the field tests. The pump began to deteriorate and required replacement after the first day of the field tests when it could no longer deliver the necessary 5 liters per minute (L/min) and could only pull 1.5 L/min. Because some of the smokes appeared to coat tube layers, the pump and manifold flows were checked after each smoke test. The flows slowly decreased throughout the day but remained within 30% of the starting flows. Since the preset flows for each tube had dropped to almost 30% of the starting values after the Field Tests were completed, the pump was again replaced before continuing with further laboratory evaluations to ensure that flows were returned to the manufacturer's preset values.

The HD and HD, HN tubes showed no false positives for any of the field test exposures. The yellow of the tubes did not change color appropriately indicating no agent present, however, the tubes discolored to a dirty yellow and a charcoal or brown ring was noticed during most of the burning material tests.

The PAE tubes did not produce any confirmed false positive responses to any of the field test interferents. The PAE tubes only remained white, indicating a false positive, for one of three bleach vapor exposure trials. The second trial against the bleach vapor produced a light yellow response so a third tube was tested against the bleach vapor that produced a cream color confirming a negative response. Exposure to diesel vapor and exposure to burning cloth smoke both produced a cream color for one of the trials and a light yellow color for the other exposure. The PAE tube exposures to the other field test interferents showed the appropriate negative yellow responses although the operator descriptions for half of all the field test exposures were light yellow, splotchy yellow and cream. Some of the blanks were also described as light yellow and splotchy light yellow. It was noted by the operators that the light yellow color of the tubes is hard to see in the sunlight.

Table 7. Results of Field Interference Tests of Civil Defense Kit™ HD only, HD, HN, and Phosphoric Acid Esters (PAE) Tubes at Ambient Conditions

Interferent	HD Only Tubes	HD, HN Tubes	Phosphoric Acid Ester Tubes
Gasoline Engine Exhaust, Idle	Negative	Negative	Negative
Gasoline Engine Exhaust, Revved	Negative	Negative	Negative
Diesel Engine Exhaust, Idle	Negative	Negative	Negative
Diesel Engine Exhaust, Revved	Negative	Negative	Negative
Gasoline Vapor	Negative	Negative	Negative
Diesel Vapor	Negative	Negative	Negative
JP8 Vapor (Jet Fuel)	Negative	Negative	Negative
Kerosene Vapor	Negative	Negative	Negative
AFFF (6%) Vapor	Negative	Negative	Negative
Clorox (6% Bleach) Vapor	Negative	Negative	Negative*
Insect Repellent (DEET)	Negative	Negative	Negative
HTH (10% calcium hypochlorite) Vapor	Negative	Negative	Negative
Burning Gasoline Smoke	Negative	Negative	Negative
Burning JP8 Smoke	Negative	Negative	Negative
Burning Kerosene Smoke	Negative	Negative	Negative
Burning Diesel Smoke	Negative	Negative	Negative
Burning Cardboard Smoke	Negative	Negative	Negative
Burning Cloth Smoke	Negative	Negative	Negative
Burning Wood Fire Smoke	Negative	Negative	Negative
Doused Wood Fire Smoke	Negative	Negative	Negative
Burning Tire Smoke	Negative	Negative	Negative

Note: Negative response reading indicates no color change for the HD tube = no agent detection. For the PAE tubes, a negative response means a color change to yellow = no agent detection.

*One out of three trials showed a false positive

6. CONCLUSIONS

Conclusions are based solely on the results observed during this testing. Aspects of the Civil Defense Kit™ system other than those described were not investigated. Three of the Kit's five types of colorimetric detector tubes were evaluated. The nerve agent tube, phosphoric acid esters (PAE) with part number 10007654-msa, was tested against both GA and GB. The HD only tube (part number 10007653-msa) and the HD, HN tubes (part number 10007652-msa) were each tested against HD.

Civilian first responders and HAZMAT personnel use Immediate Danger to Life or Health (IDLH) values to determine levels of protection for selection of personal protective equipment during consequence management of an incident. The PAE tubes demonstrated detection of GA and GB to meet the IDLH detection limits. However, none of the tubes were able to meet the much lower Airborne Exposure Limit (AEL) detection requirements for GA, GB, or HD. The minimum detection limit (MDL) of the Civil Defense Kit™ tubes tested was slightly above the current Joint Service Operational Requirement (JSOR) for point sampling detectors for the agents HD and GA. The PAE tube was able to accurately respond to GB at the JSOR value. The Civil Defense Kit™ response time requires at least 7.5 minutes for sampling, activation and color development of the tubes.

None of the tube types would perform in the cold temperature of 5^{o}C. The HD only tubes and the HD, HN tubes did not respond to HD at concentrations up to three times the MDL values. In the cold temperature, the PAE tubes produced false positive indications on blank runs in the absence of CW agent.

In addition, the PAE tubes produced false positive response on blank runs in the high temperature tests at 40^{o}C. However, at 40^{o}C the HD only tube response was much more pronounced and HD detection was seen at half the previously determined MDL value. The HD, HN tube responded correctly at the MDL for HD at 40^{o}C.

High humidity tests above 80% RH had adverse effects on the tubes for agent detection except against GB. The HD only tubes failed to detect HD at high humidity even at much higher than the determined MDL concentration levels. During the high humidity tests the PAE tubes gave a positive response to GA at three times the MDL values and the HD, HN tube required approximately two times the MDL concentration for HD detection.

It appears that low humidity (<5% RH) at ambient temperatures had a slight adverse effect on HD detection for the HD, HN tube and on GA detection for the PAE tube. Both tube types required a slightly higher than MDL concentration for the respective agent for positive detection response. Low humidity had no effect on the HD only tube or the PAE tube for detection of GB at the respective MDL values.

There was a large amount of subjectivity in determining the color development for both HD only and HD, HN tubes or the lack of color development for the PAE tubes indicating positive responses near the CW agent threshold levels. For example, several people would observe a color change and give different interpretations of tube response. Positive detection responses, however, were more distinguishable with increasing CW agent vapor concentrations higher than the determined MDL. The intensity and broadness of the color band increased as the concentration increased.

The Civil Defense Kit™ colorimetric tubes show promise for use in emergency response situations to detect the presence of CW agent vapors because they are relatively inert to potential interference. Field interference and laboratory interference evaluations did not produce false positive responses. The tubes retained CW agent detection capability in the presence of the potential interference substances tested in the laboratory.

The inability to use the PAE, HD only, and HD, HN tubes in cold temperatures is a limitation of concern. At 5°C, the HD tubes cannot detect HD and the PAE tubes produced false positive responses to blanks. It appears that the cold temperature suppressed the chemical reactions required for these tubes to function properly. Further testing is required for conclusive results at high temperature for the PAE tubes since the PAE tubes produced several false positive responses on blank tubes and inconsistent GB detection at 40°C. The storage temperature requirement for the tube set of less than 25°C (77°F) might be an area of concern for the user as it suggests that the tubes are subject to deterioration at higher temperatures.

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Appendix
Glossary

AEL	Airborne exposure limit
AFFF	Aqueous film forming foam, used for fire fighting
AR	Army regulation
ATT	Applied Test Team
CASARM	Chemical agent standard analytical reference material
CW	Chemical Warfare
DOD	Department of Defense
DP	Domestic Preparedness
ECBC	U.S. Army Edgewood Chemical Biological Center
FPD	Flame photometric detector
GA	Tabun, a CW agent
GB	Sarin, a CW agent
GD	A CW agent
GF	A CW agent
GP	A CW agent
HAZMAT	Hazardous materials
H	A CW agent
HD	Mustard, a CW agent
HN	A CW agent
HTH slurry	Calcium hypochlorite solution, a chlorinating decontaminant for CW agents
IDLH	Immediate danger to life or health
IPE	Individual protective equipment
JP8	Jet propulsion fuel
JSOR	Joint Service Operational Requirements for detectors
L	Lewisite, a CW agent
L/min	Liters per minute
MINICAMS®	Trade name for a chemical agent detector, the “Miniature Continuous Air Monitoring System.”
MDL	Minimum detectable level
mg/m^3	Milligrams per cubic meter,
mL/min	Milliliters per minute
NBC	Nuclear, biological and chemical
PAE	Phosphoric acid esters
PCT	Pre-concentrator tube
ppm	Parts per million
RH	Relative humidity
SCBA	Self-contained breathing apparatus
TWA	Time-weighted average
VX	A CW agent